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				<b>5b. GRANT NUMBER</b>		
				<b>5c. PROGRAM ELEMENT NUMBER</b>		
<b>6. AUTHOR(S)</b>				<b>5d. PROJECT NUMBER</b>		
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## **FINAL TECHNICAL REPORT**

### **Thermal Properties of Contemporary and Conventional Gutta Percha Materials Used in Root Canal Treatment**

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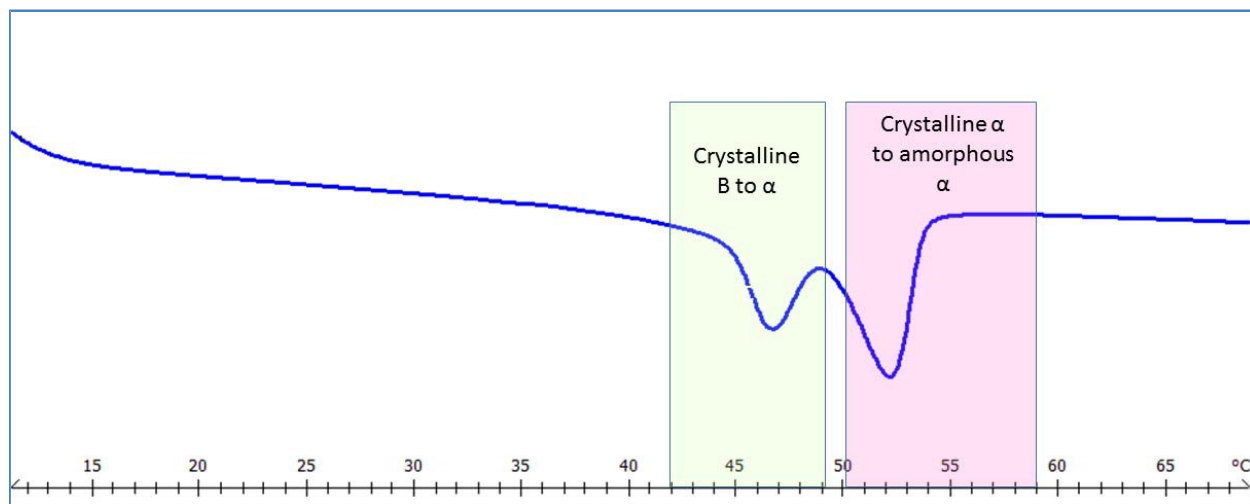
## Gutta-Percha Analysis Final Report

Dental gutta-percha is the most widely used dental material for the obturation of root canal systems and has been used for over 100 years.<sup>1</sup> The gutta-percha polymer used in dental gutta-percha cones is *trans*-1,4-polyisoprene that is obtained from the processing of latex from the Sapotaceae tree family.<sup>2</sup> Commercially-available dental gutta-percha cones are heterogeneous materials with the polyisoprene component paradoxically not being the main constituent. Zinc oxide is actually the main constituent comprising roughly 66 – 84 percent between some marketed products while the amount of gutta-percha ranges between 14 and 20 percent.<sup>3</sup> The remaining constituents of dental gutta-percha cones also vary depending on manufacturer and consist of metal sulphates (for enhanced radiopacity), waxes/resins, and coloring agents.<sup>3</sup>

The composition, mechanical and thermal properties of dental gutta-percha have been extensively described in the scientific literature.<sup>1-22</sup> The gutta-percha polymer, *trans*-polyisoprene, can exist in two crystalline and/or stereochemical forms that are known as  $\alpha$  (alpha) and  $\beta$  (beta). Naturally occurring gutta-percha exists in the  $\alpha$  crystalline phase, and if  $\alpha$ -gutta-percha is cooled at a rate greater than 0.5 °C per hour it can be transformed into the  $\beta$  phase.<sup>7</sup>

Gutta-percha processed for dental use has been described as largely existing in the  $\beta$  semi-crystalline state,<sup>8,18</sup> although it has been reported that some manufacturers claim to produce a dental gutta-percha that is in the  $\alpha$  phase.<sup>18</sup> When the  $\beta$ -form of pure gutta-percha is analyzed over increasing temperature, two endothermic peaks usually occur (Figure 1).

**Figure 1. Gutta-percha thermal enthalpy curves**



The first endothermic peak (approximately between 42 and 49 °C) annotates the transformation of the crystalline  $\beta$ -phase to the crystalline  $\alpha$ -phase. The second peak (approximately between 50 and 59 °C) is due to the conversion of the  $\alpha$  crystalline form to an amorphous gutta-percha.<sup>6,18</sup> It should be noted that these temperatures for commercial endodontic gutta-percha will be slightly different, as these temperature values are for pure gutta-percha.

With the increased number endodontic gutta-percha materials being marketed for different thermal obturation techniques, some confusion exists for clinicians in choosing the proper obturation material for a

particular clinical technique. Furthermore, some literature has suggested that not all commercially available endodontic gutta-percha materials exist in the same semi-crystalline phase<sup>18</sup> while other studies suggest that some materials are not stable at the higher temperatures that may be associated with warm vertical condensation techniques.<sup>20</sup> Additionally, other non-gutta-percha materials that have similar handling characteristics have been introduced as resin-based materials. The purpose of this research was to evaluate the thermal characteristics and stability of 23 current commercially-available endodontic obturation materials.

## **Methods and Materials**

The gutta-percha products evaluated are listed in Table 1. Representative samples were obtained from the terminal three millimeters of ISO Size 40 obturation cones and/or cartridges. Samples were placed in 40-micron aluminum differential scanning calorimetry (DSC) crucibles and sealed. They were placed into a DSC unit (DSC 1, Mettler Toledo, Columbus, OH, USA) and subjected to a first scan (annotated as “A”) that consisted of a 25 to 70 °C scan at a rate of 5 °C/min after which the temperature was rapidly increased up to 230 °C. At the end of this first scan the sample was immediately removed and placed on an aluminum block at ambient temperature. Immediately a second scan (annotated as “B”) was repeated from 25 to 70 °C at a rate of 5 °C per minute. The initial portion of the first “A” scan was used to identify the thermal phases of the commercial gutta-percha samples as received, and then to simulate the thermal challenge that gutta-percha would encounter during canal obturation. The following second “B” scan only contained the 25 to 70 °C scan to observe phase changes possibly caused by the previous high temperature challenge. This two-scan process for each sample was then repeated twice (hence annotated as “A2, B2, A3, B3”) to observe any changes possibly encountered by repeated high heat exposure during obturation. The enthalpy of the different phases and as well as peak temperatures were analyzed using thermal analysis software (Star<sup>e</sup>, Version 10.1, Mettler Toledo). Ten samples were analyzed for each material investigated with all enthalpies normalized as to specimen weight. Mean enthalpies for each scan were determined and compared within each material using statistical software (SPSS ver 18.0, IBM, Armonk, NY, USA) using Kruskal-Wallis after subjecting the data to Levene and Brown-Forsythe analysis for equality of variance and data distribution.

## **Results**

The graphical results of the mean thermal curves are displayed in Figures 2- 24.

Figure 2. Calamus

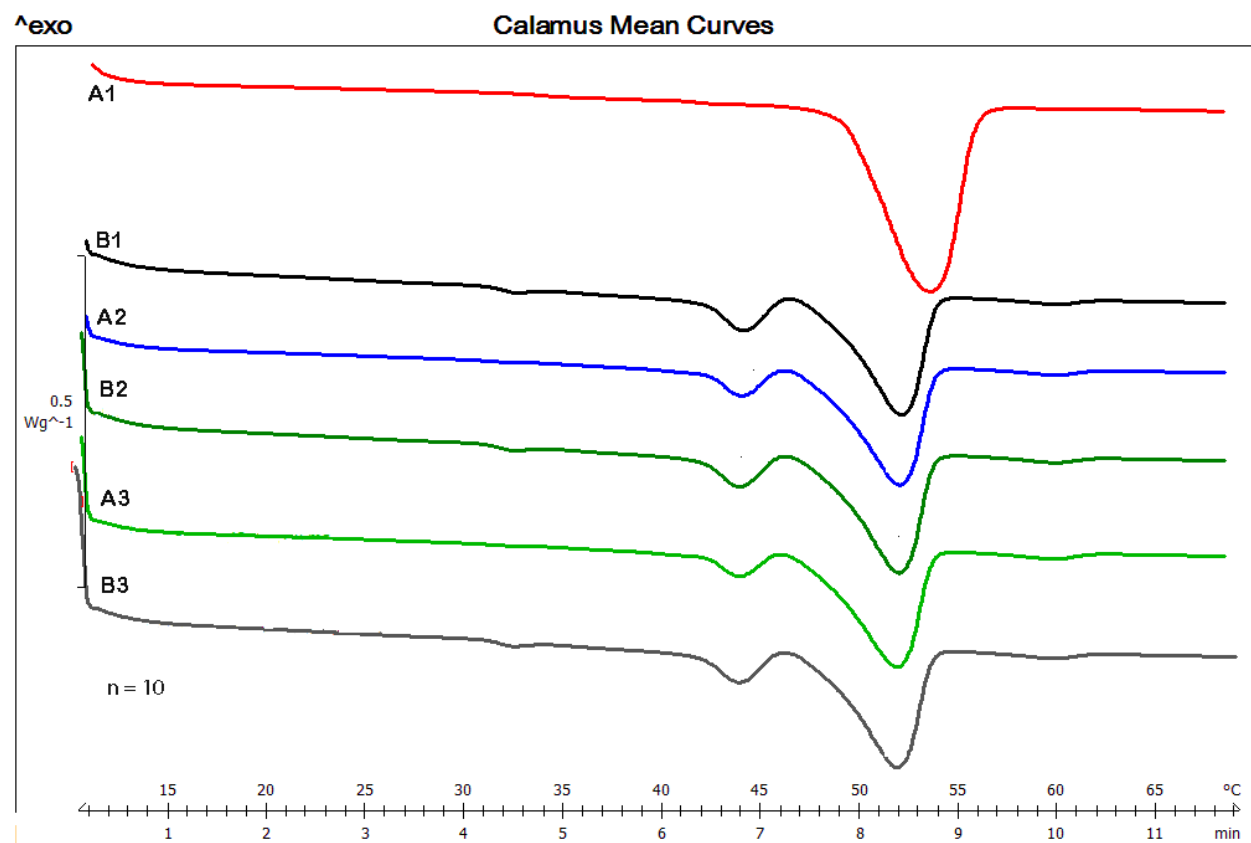


Figure 3. Diadent

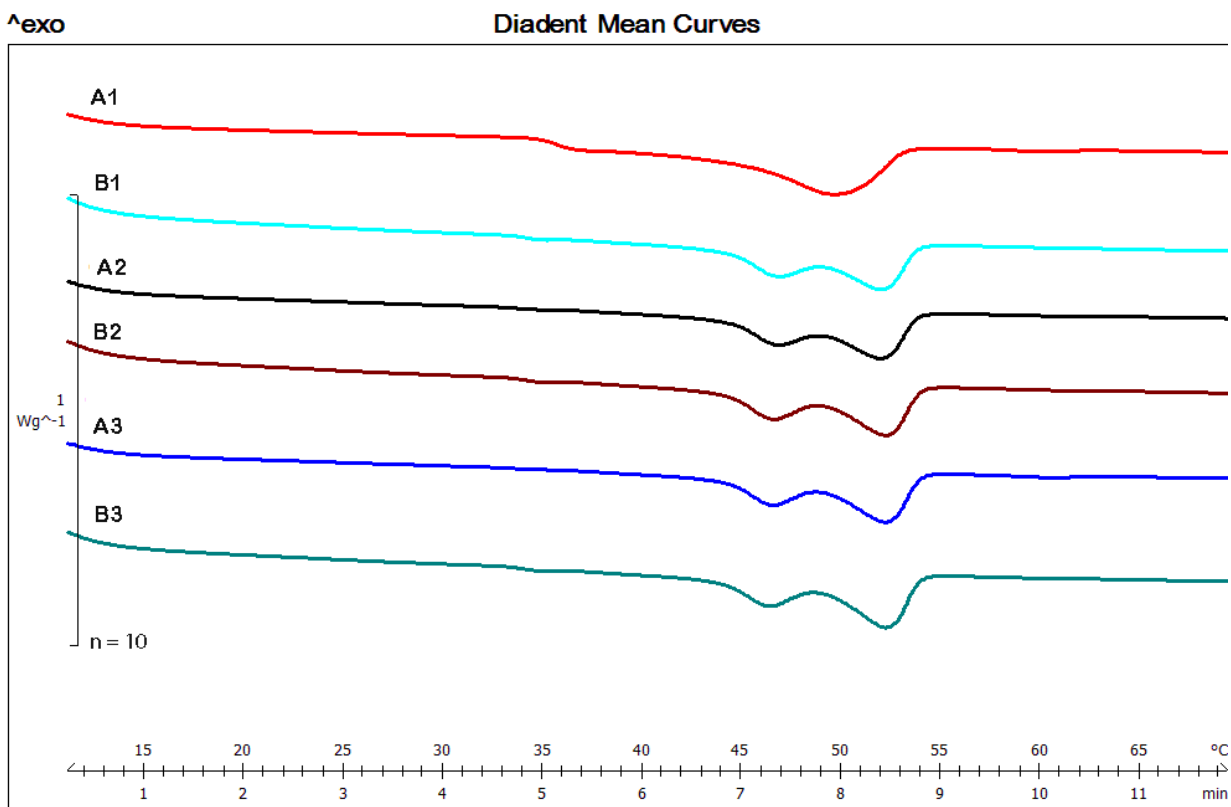


Figure 4. Elements HB

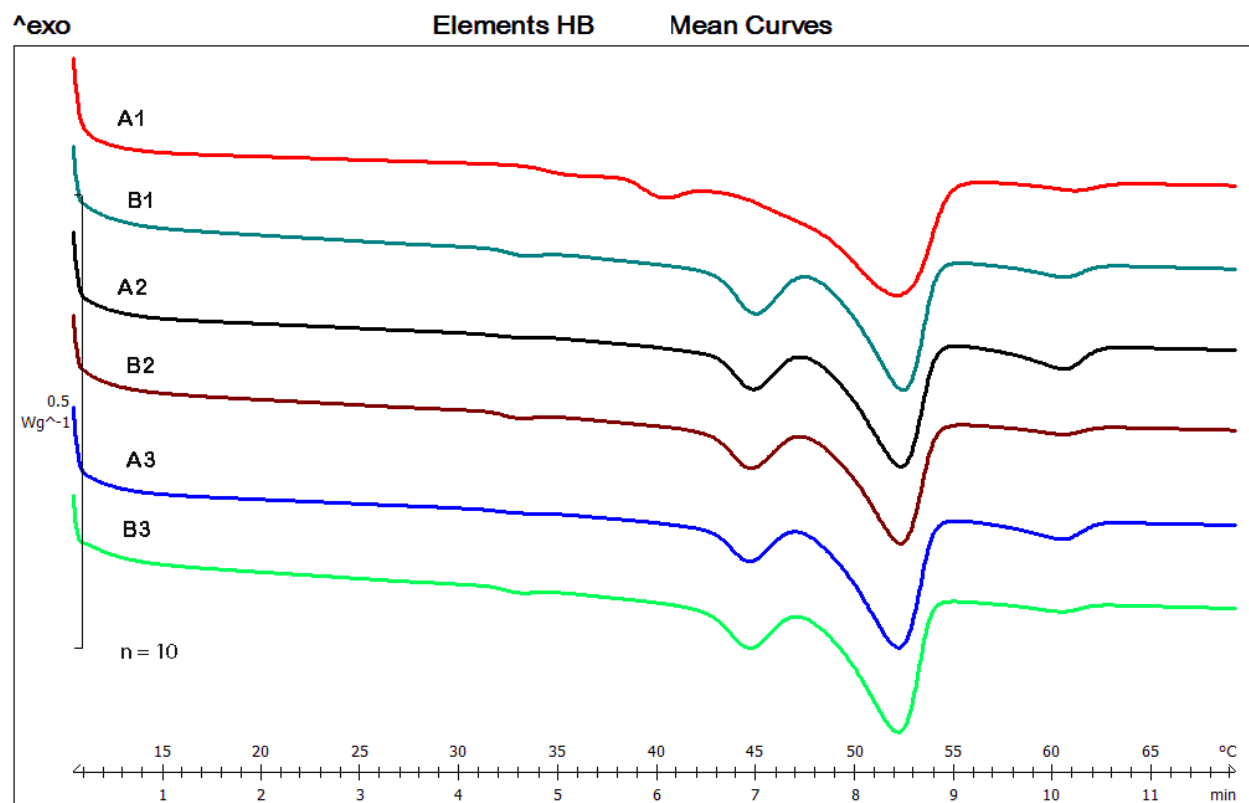


Figure 5. EndoRez

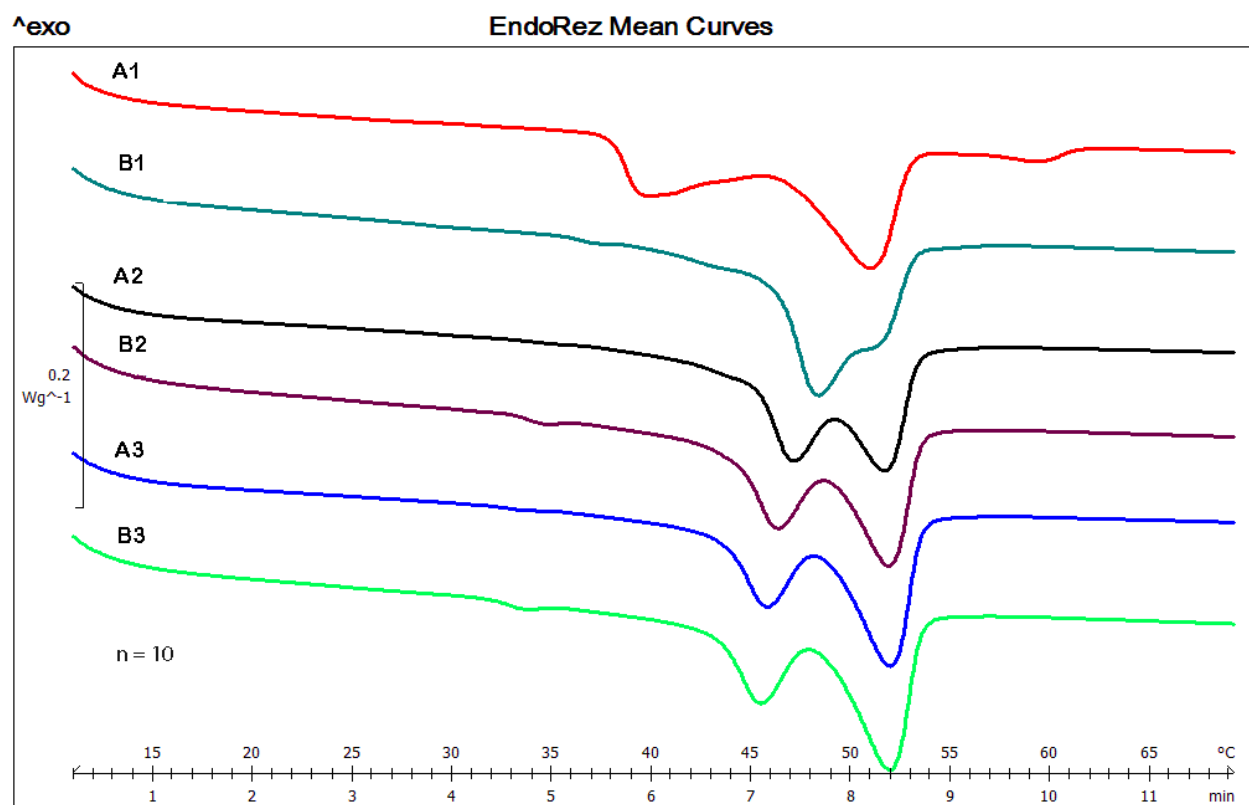




Figure 6. EndoSequence

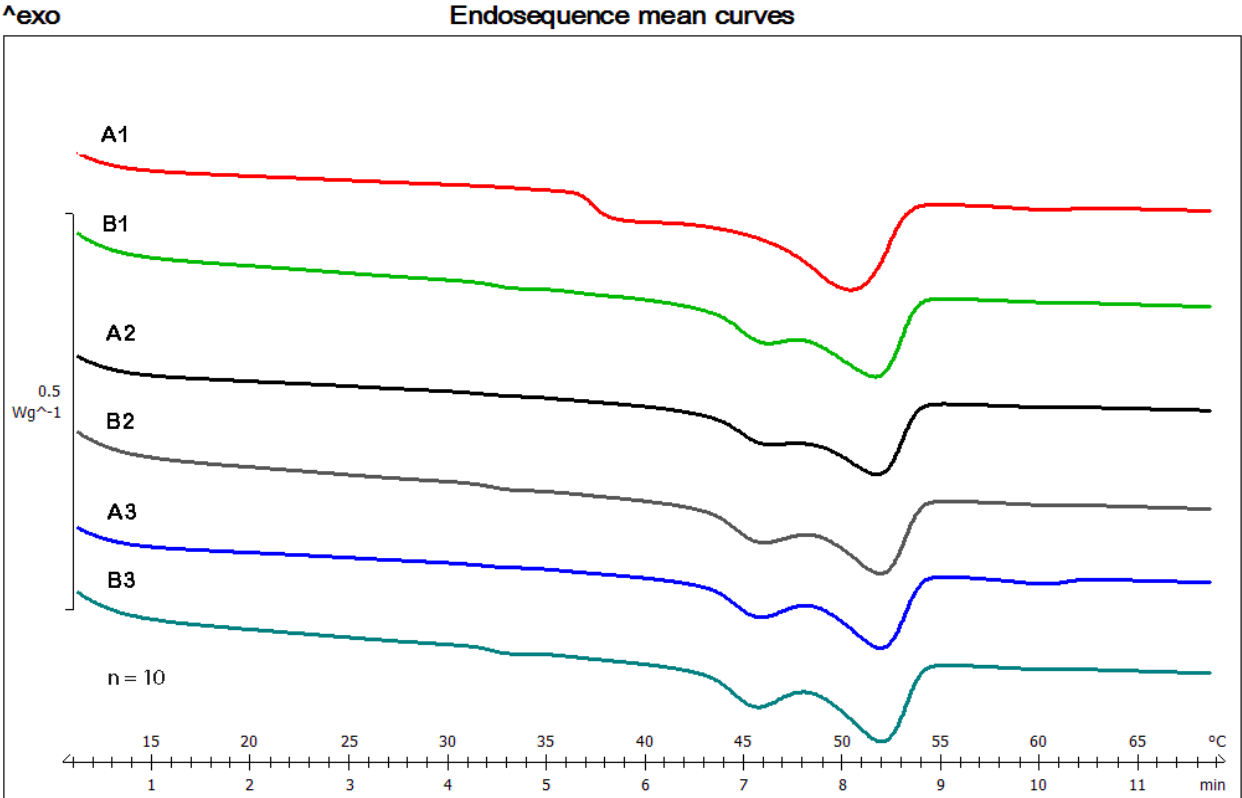


Figure 7. Gutta Core

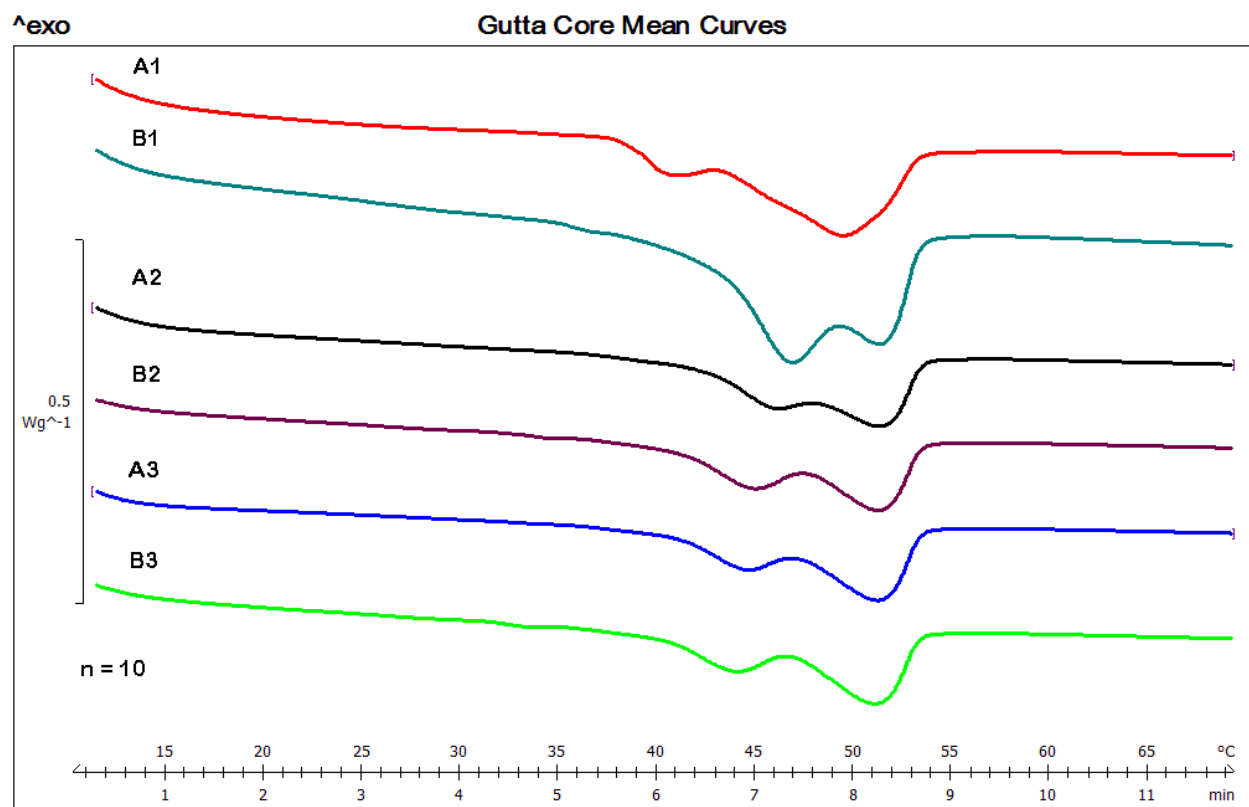


Figure 8. Gutta Flow

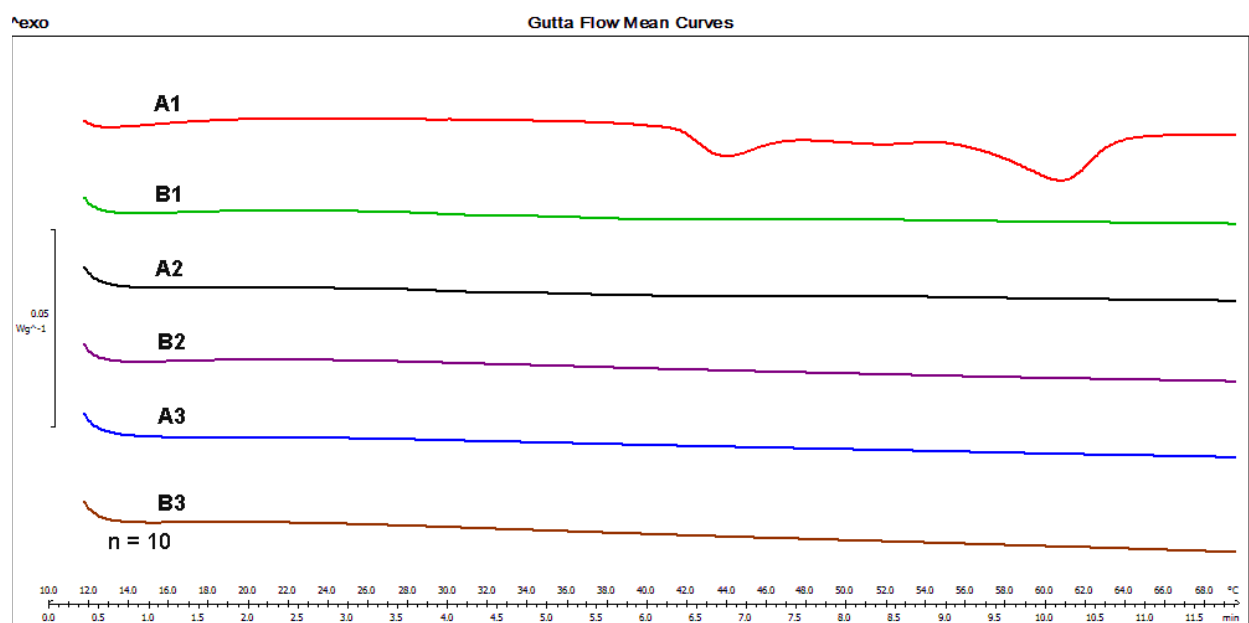


Figure 9. K3

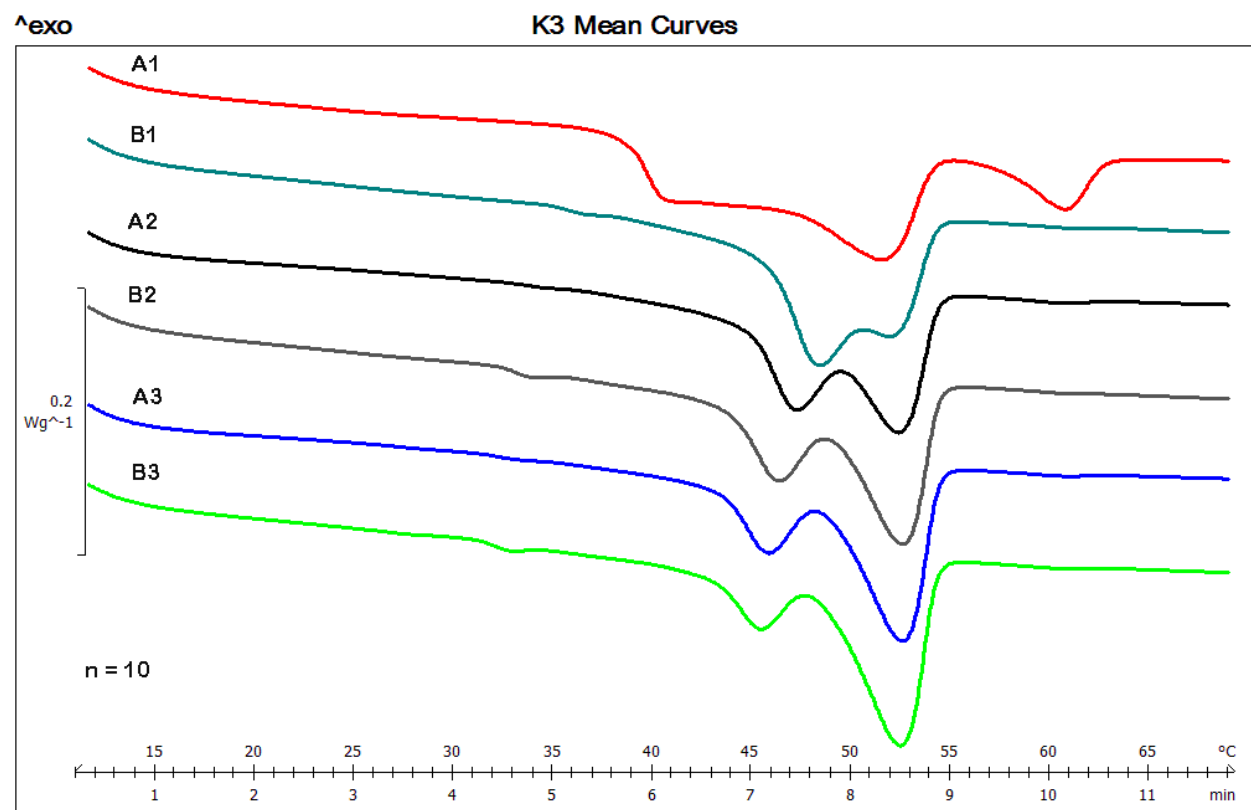


Figure 10. Lexicon

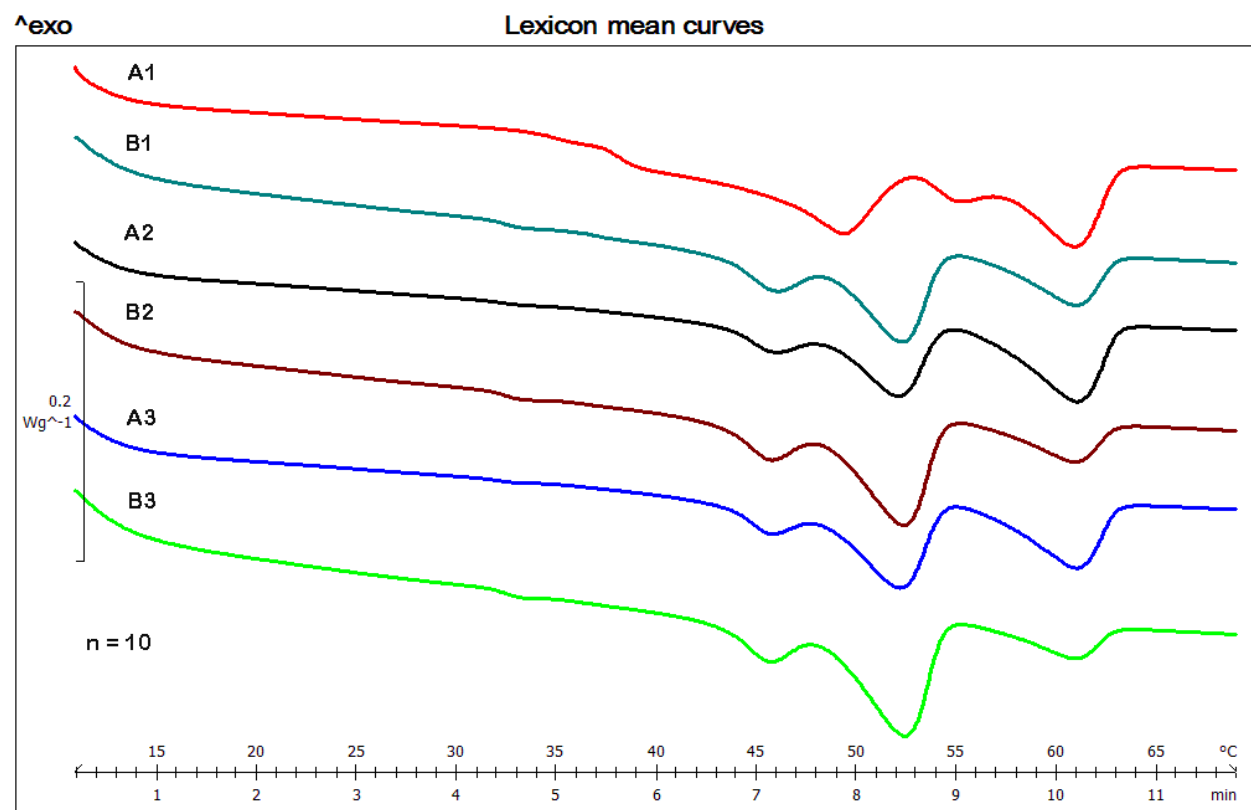


Figure 11. Microseal

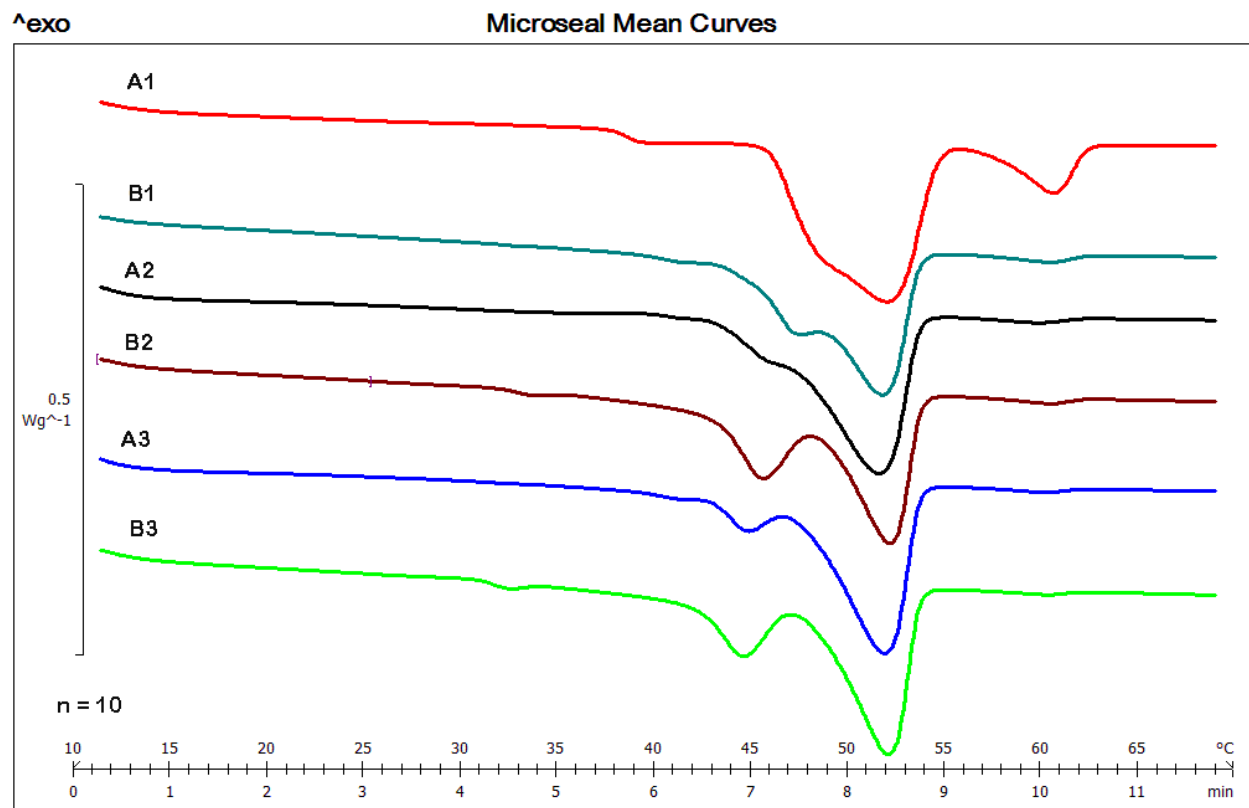


Figure 12. Obtura Flow

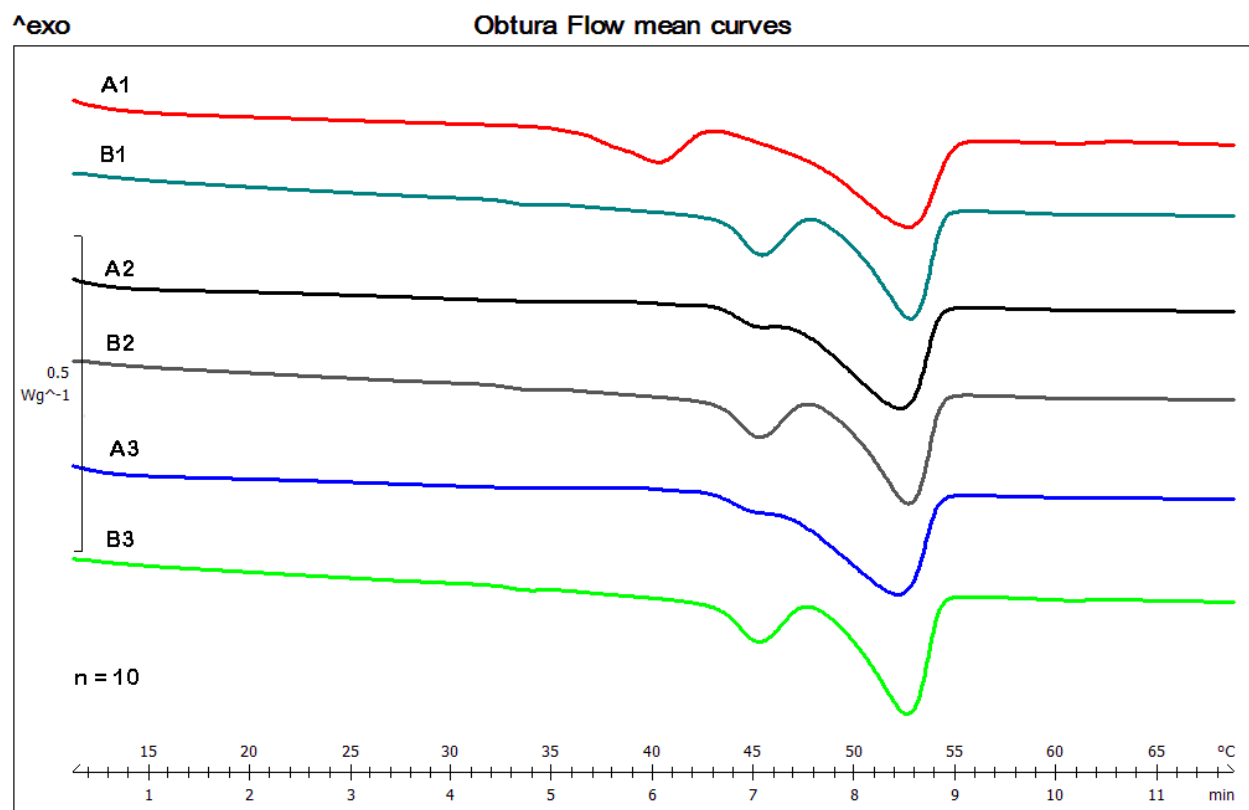


Figure 13. Obtura

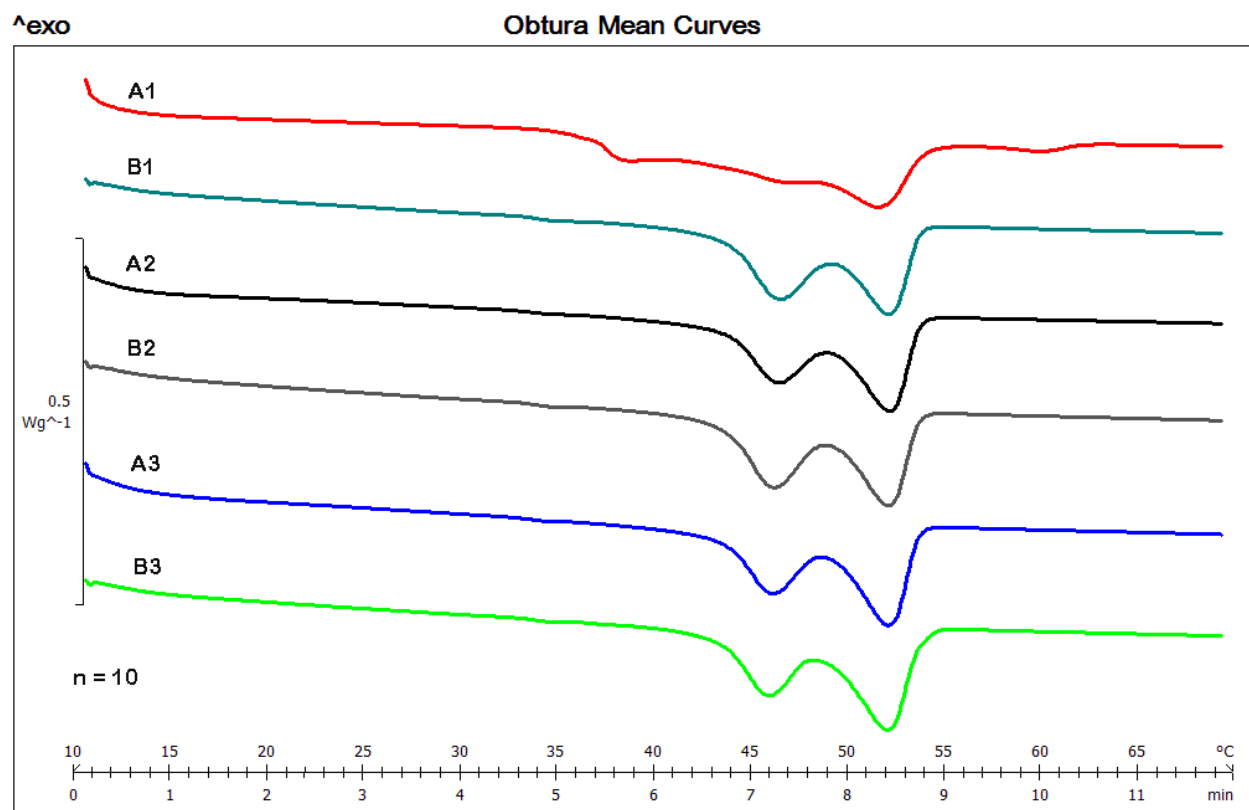


Figure 14. Real Seal

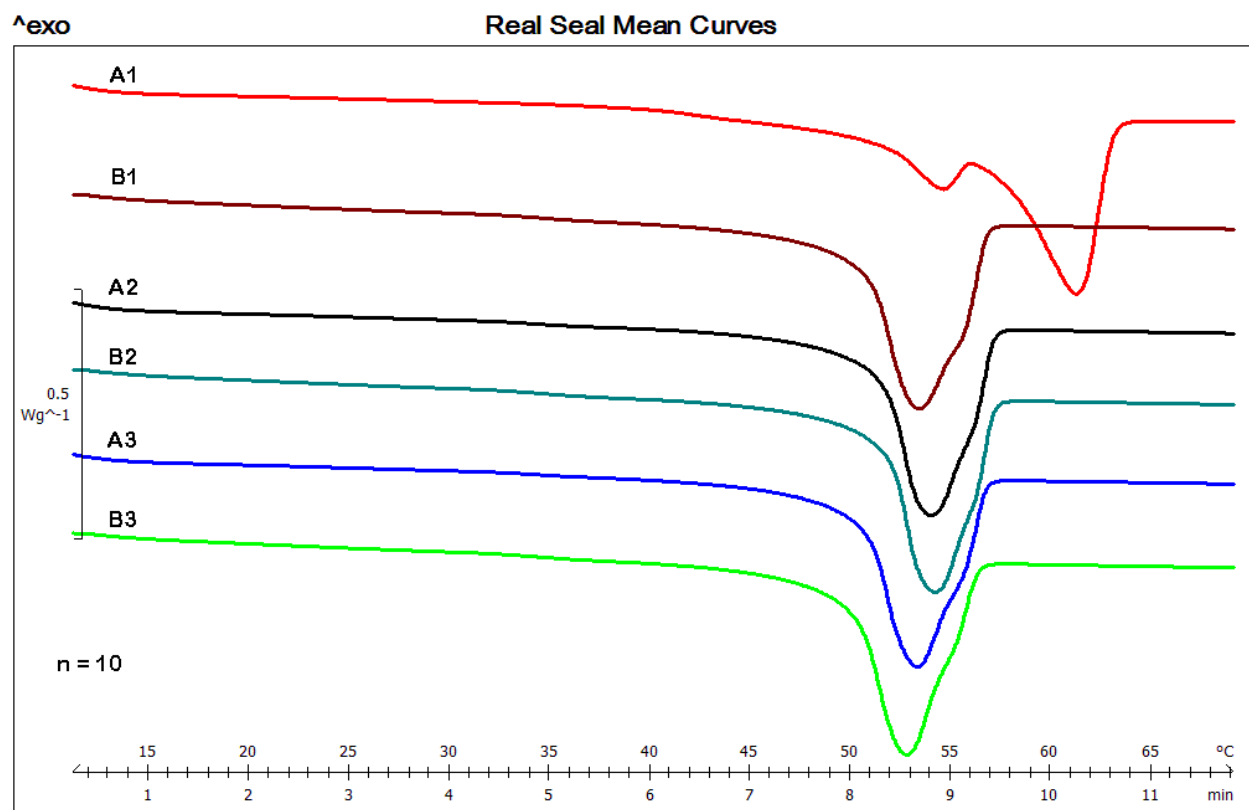


Figure 15. Resilon



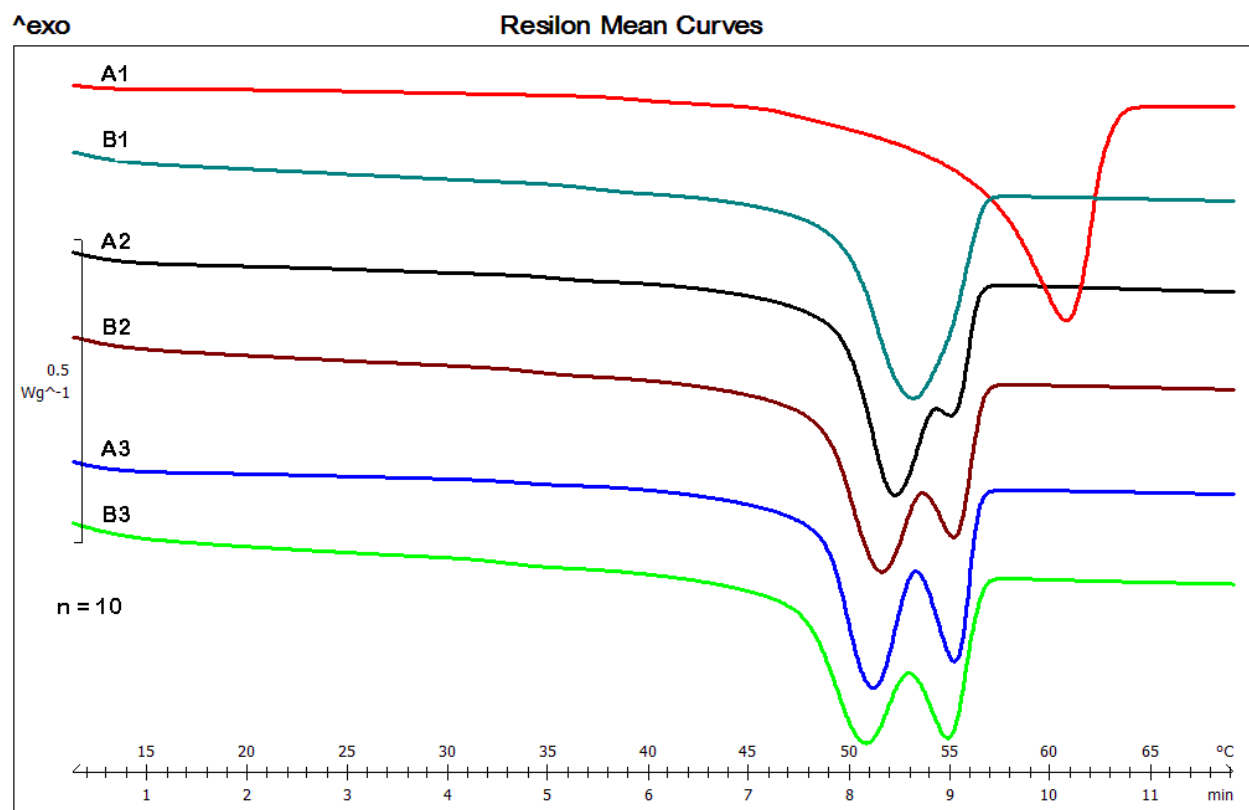


Figure 16. Schein Accessory Points

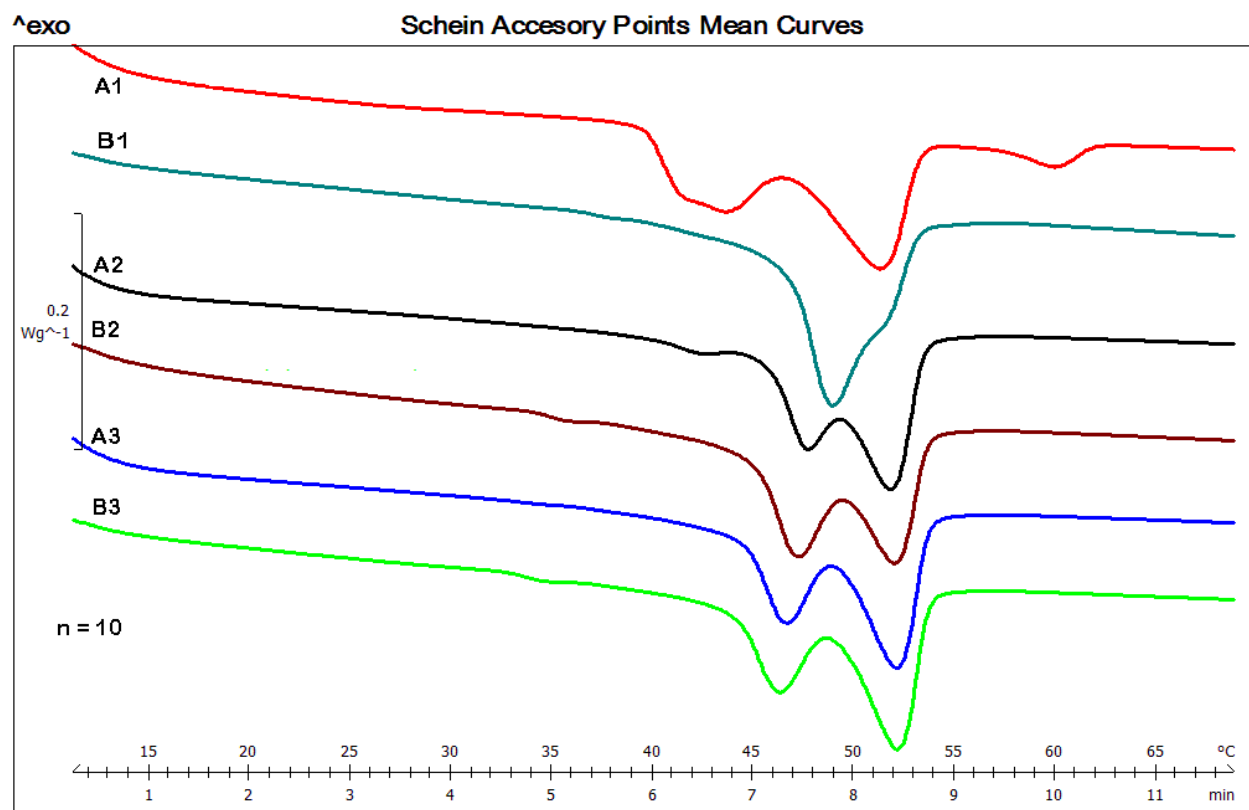


Figure 17. Simplifill

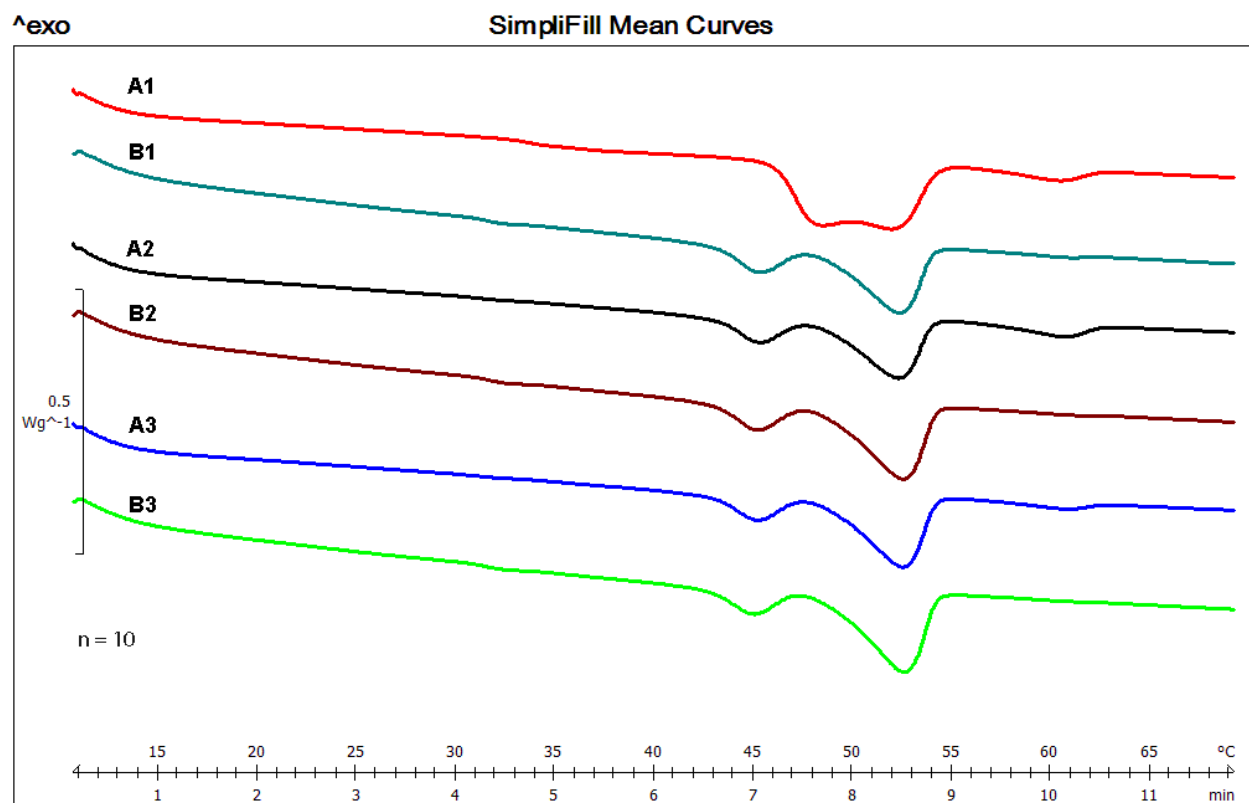


Figure 18. Smart Endo

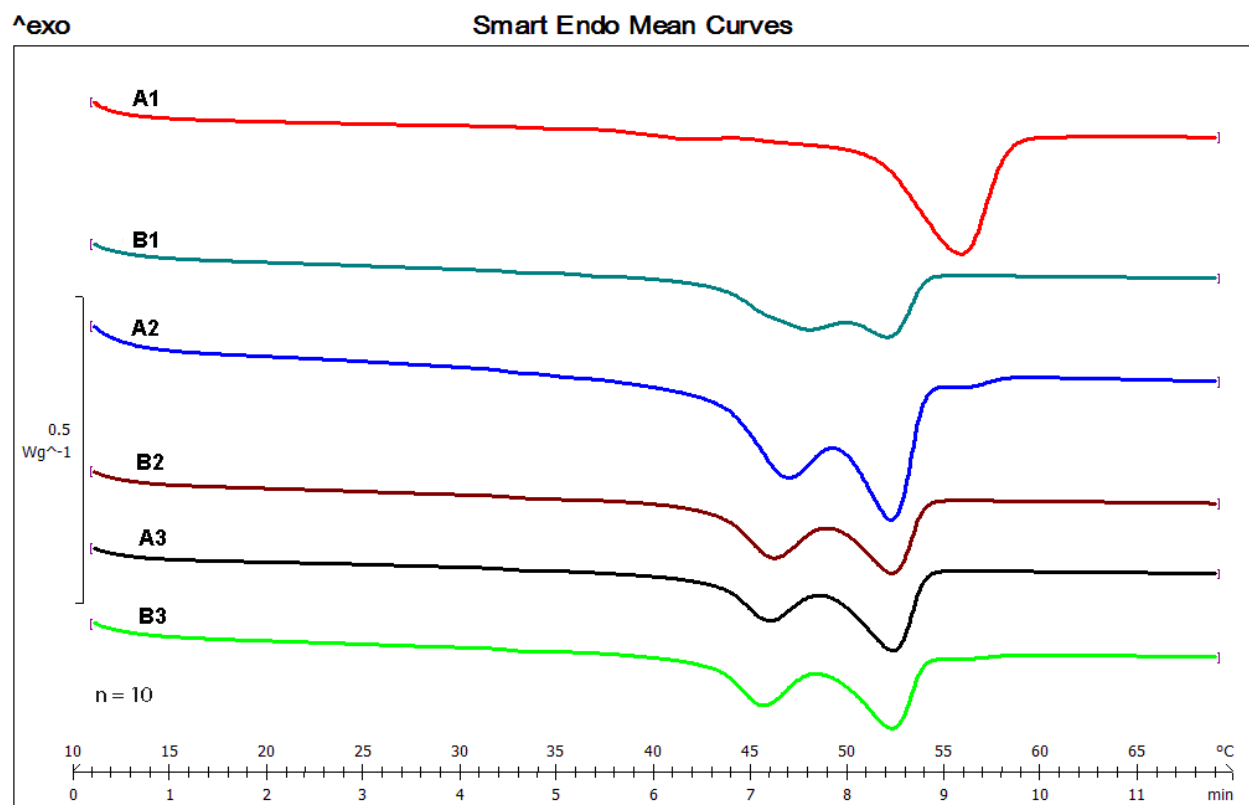


Figure 19. SpectraPoint

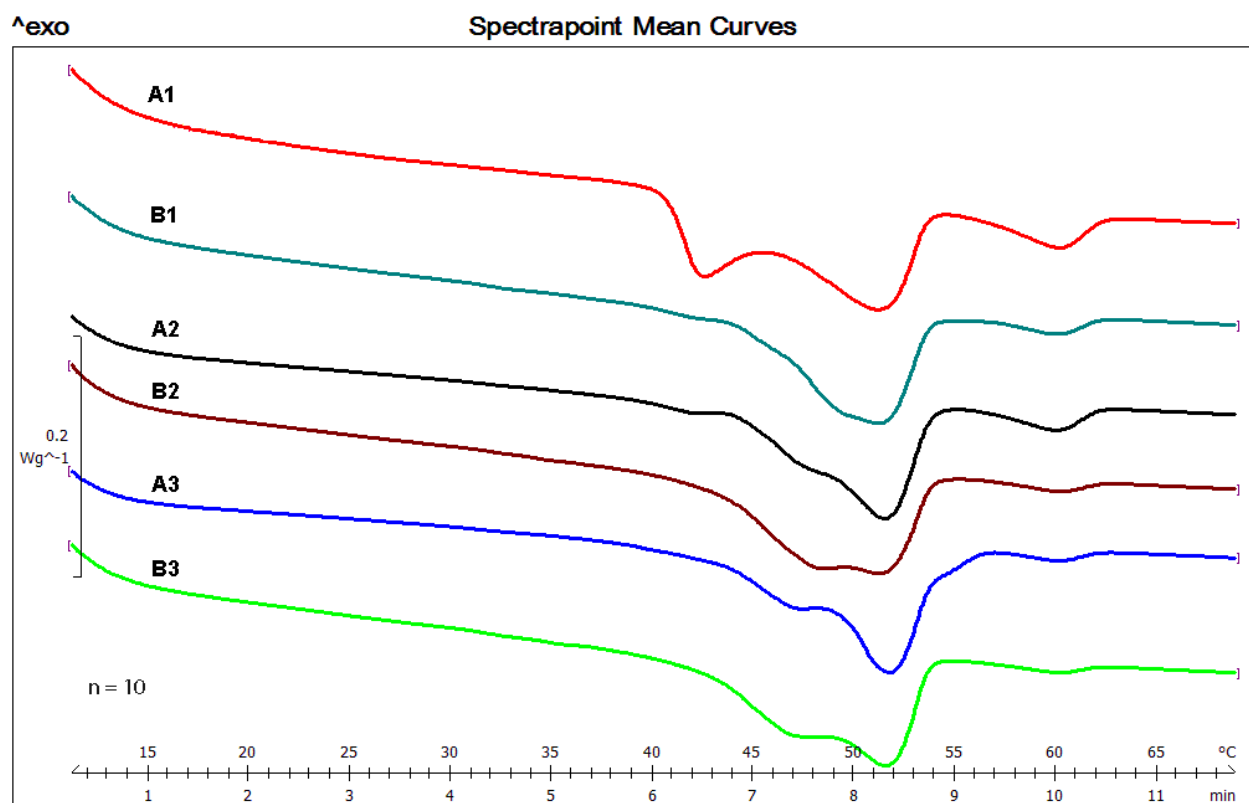


Figure 20. SuccessFill

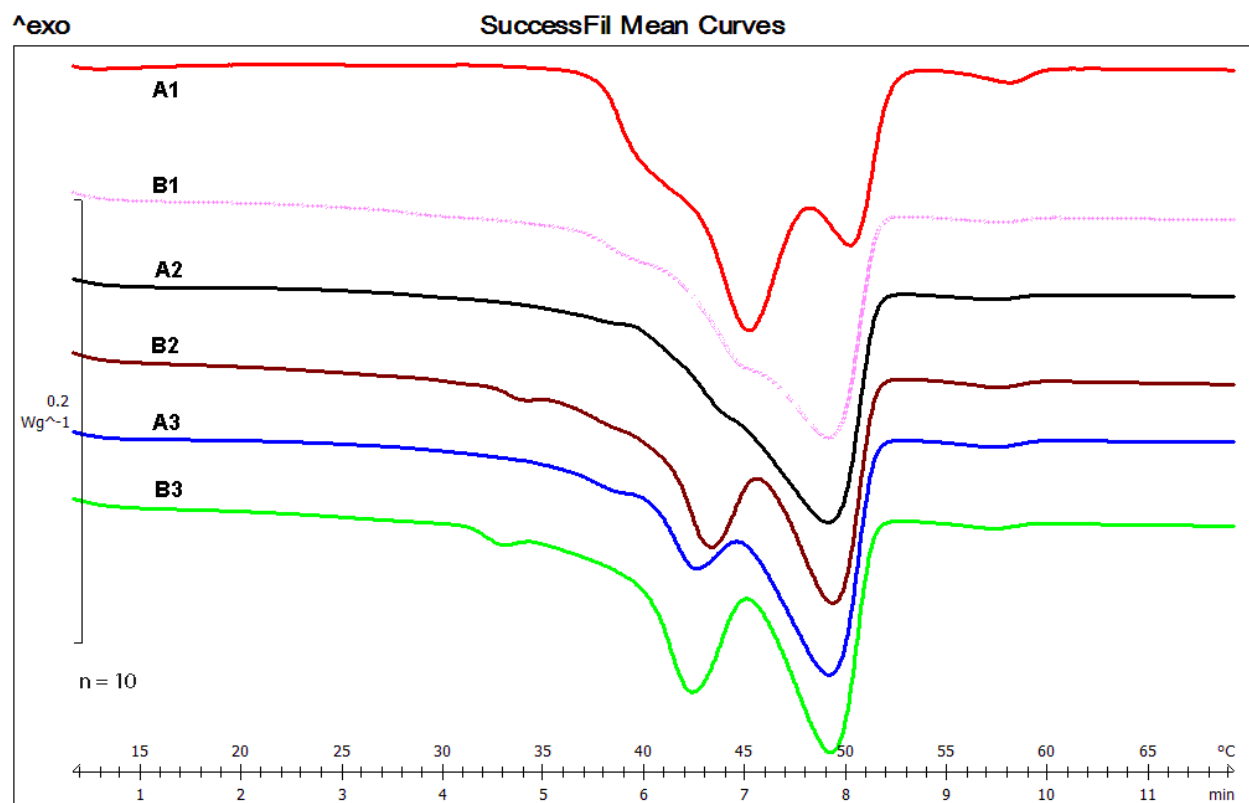


Figure 21. Thermafill

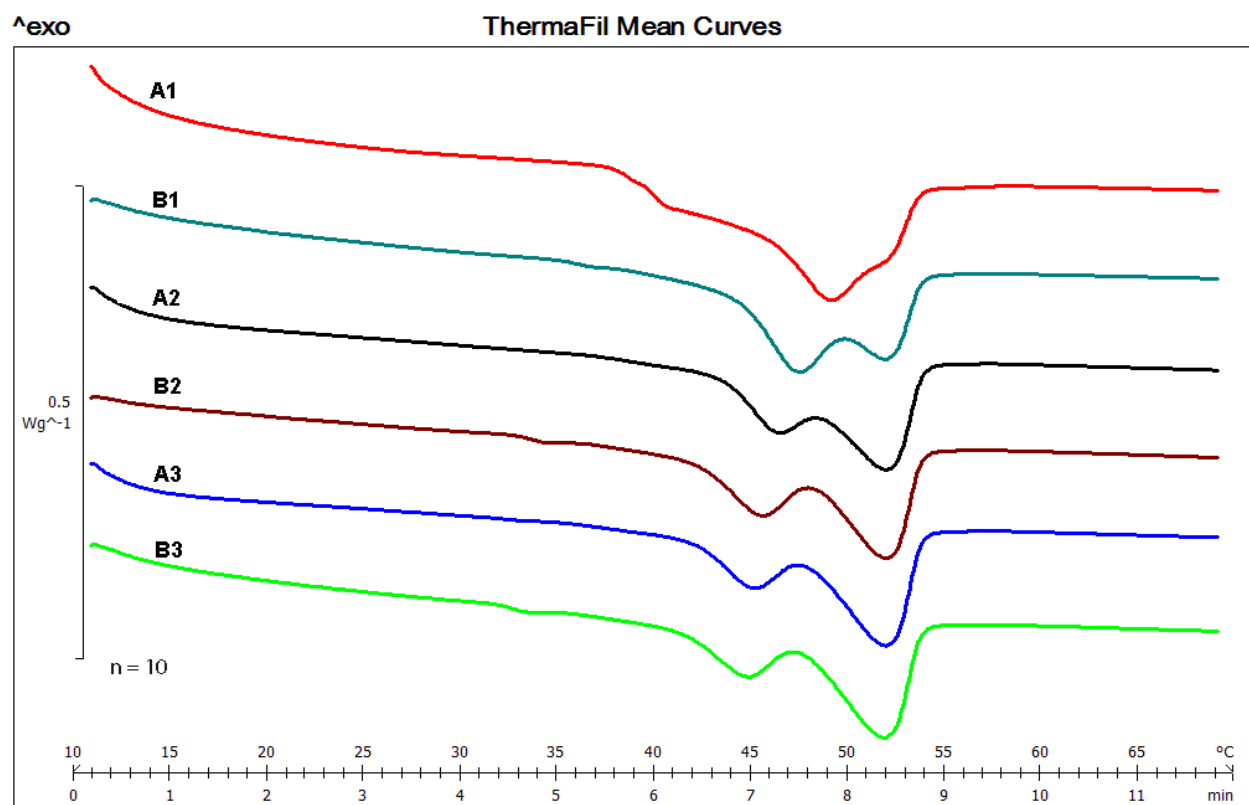


Figure 22. UltraFil EndoSet

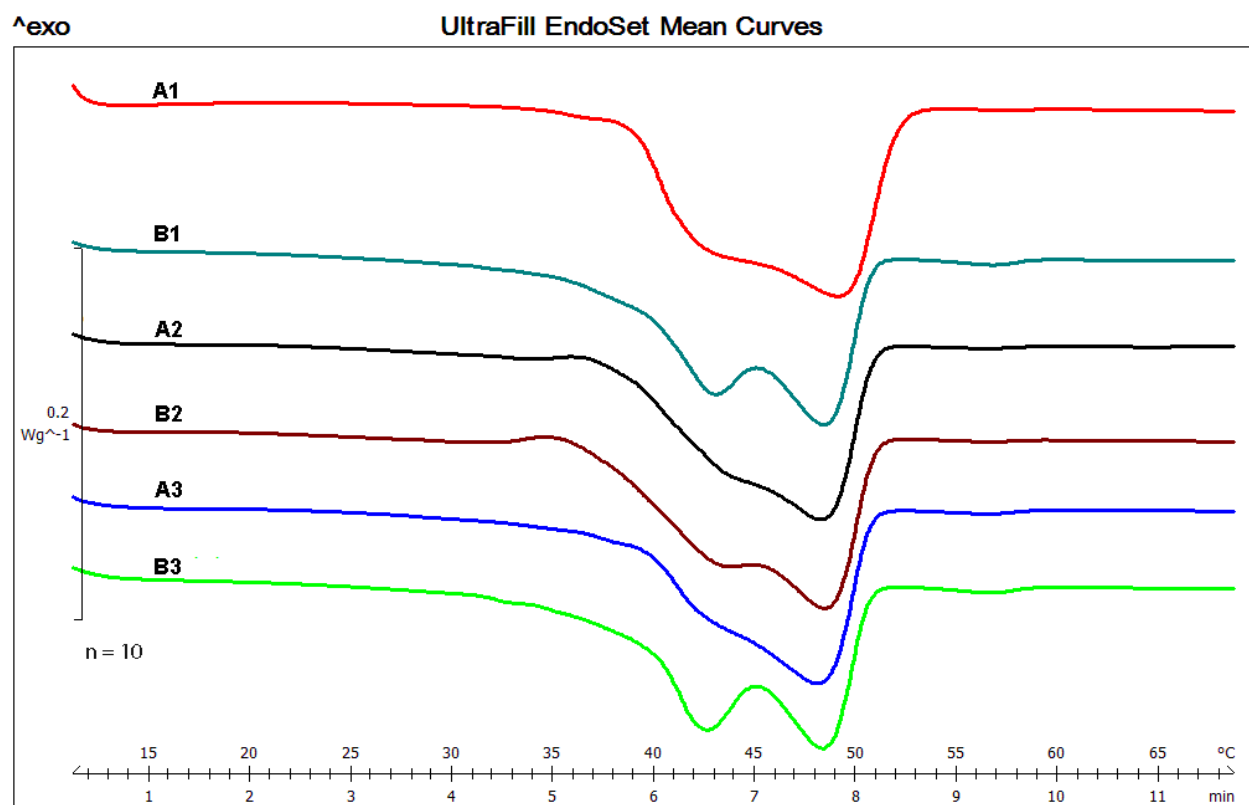


Figure 23. UltraFil FirmSet

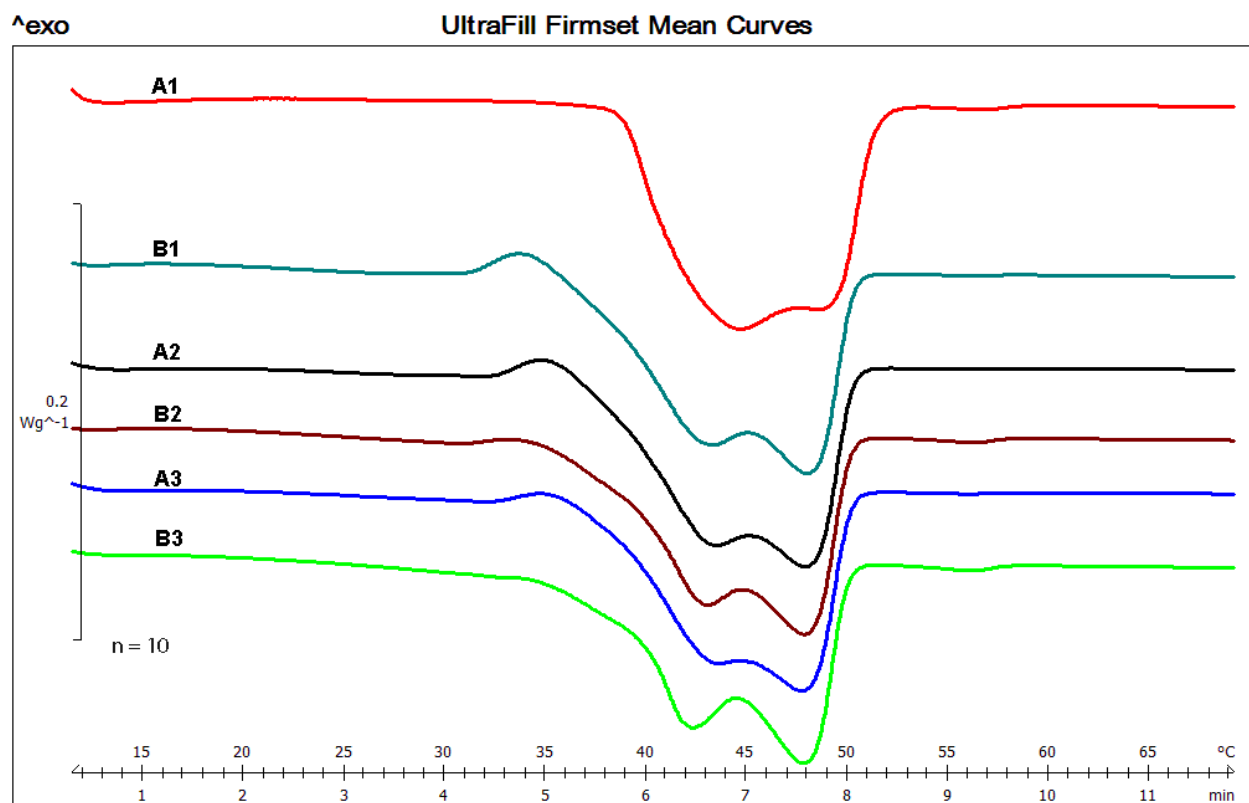
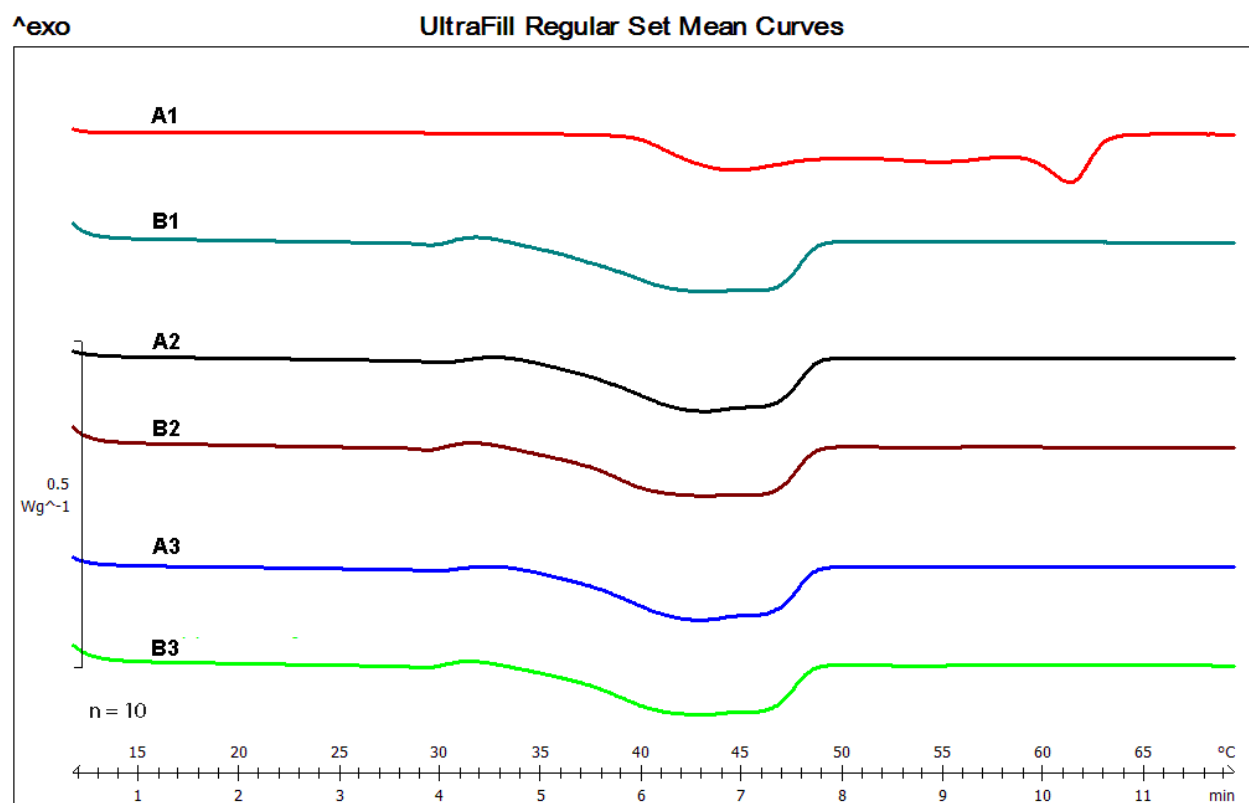


Figure 24. UltraFill Regular Set



Typically, upon the first thermal evaluation (Run A1), most of the materials demonstrated a broad endothermic enthalpy curve. Subsequent thermal evaluations usually depicted more definitive demonstrations of the alpha and beta thermal signatures, whose thermal enthalpies were then determined by integration of the respective curves. The mean thermal results for both the beta- and alpha thermal integrations are displayed in Tables 2 and 3, respectively.

**TABLE 2. Beta-Phase Mean Thermal Enthalpy (J/g)**

Material	RUN	A1	B1	A2	B2	A3	B3
Calamus	Mean	*	-2.56 A	-1.90 B	-2.52 A	-1.66	-2.56 A
	SD		0.31	0.23	0.14	0.13	0.19
DiaDent	Mean	*	-3.79 A	-3.65 A	-3.71 A	-3.48 A	-3.59 A
	SD		1.37	1.29	1.32	1.24	1.27
Elements HB	Mean	-1.07 A	-2.97 B	-2.16 AC	-2.65 BC	-2.24 AC	-2.70 BC
	SD	1.19	0.28	0.41	0.97	0.52	0.99
EndoRez	Mean	-4.07 A	-0.96 B	-5.39 A	-5.02 A	-4.23 A	-4.30 A
	SD	1.04	2.39	1.03	0.77	0.57	0.46
EndoSequence	Mean	*	-3.22 A	-3.48 A	-3.71 A	-3.01 A	-3.48 A
	SD		1.73	1.40	0.71	1.13	0.54
GuttaCore	Mean	-2.00 A	-6.10 B	-4.19	-3.48 B	-3.64	-3.78
	SD	0.84	0.87	0.55	2.89	0.42	0.46
K3	Mean	-2.28 A	-4.32 ABC	-4.92 B	-4.31 B	-3.76 BC	-3.21 AC
	SD	1.01	2.98	0.24	0.52	0.82	0.75
Lexicon	Mean	-5.95 A	-2.25 B	-1.62 B	-2.03 B	-1.62 B	-1.95 B
	SD	0.45	0.80	0.35	0.38	0.26	0.40
Microseal	Mean	-0.64 A	-3.93 C	-0.33 A	-4.97 D	-2.22 B	-4.33 C
	SD	0.11	0.60	1.05	0.32	1.01	0.31
Obtura	Mean	-5.75 AB	-5.50 A	-5.12 AB	-5.32 AB	-4.86 AB	-4.55 AB
	SD	2.56	0.47	0.41	0.30	0.38	1.62



Obtura Flow	Mean	*	-1.96 A	-0.27 B	-1.93 A	-0.13 B	-1.87 A
	SD		1.31	0.43	1.33	0.28	1.32
Real Seal	Mean	*	*	*	*	*	*
	SD						
Resilon	Mean	*	-1.24 A	-13.72B	-15.76 B	-15.77 B	-12.96 B
	SD		3.91	7.35	0.88	0.74	4.69
Schein Accesory Points	Mean	-4.29 A	-0.71 B	-3.69 ABC	-5.49 C	-4.44 A	-4.58 A
	SD	0.27	2.24	1.98	0.48	0.19	0.42
Simplifil	Mean	-5.29 A	-2.69 B	-2.22 B	-2.59 B	-2.25 B	-2.47 B
	SD	0.75	0.41	0.31	0.23	0.23	0.34
SpectraPoint	Mean	-3.24 A	-1.51 A	-2.13 A	-2.19 A	-3.00 A	-4.04 A
	SD	0.51	2.63	2.17	2.35	1.63	1.60
SmartEndo Medium	Mean	-0.66 A	-4.83 BC	-5.61 BC	-5.41 B	-4.87 BC	-4.43 BC
	SD	1.82	2.86	0.73	0.35	0.30	1.58
Thermafil Plus	Mean	-1.68 A	-6.38 B	-4.02 CD	-4.58 C	-3.66 CD	-3.70 CD
	SD	1.33	0.46	0.37	0.45	0.40	0.50
Successfil	Mean	-8.69 A	-4.18 BC	-2.82 D	-4.97 BC	-4.05 BC	-5.05 B
	SD	0.37	1.01	1.09	1.33	0.62	0.35
Ultrafil Endoset	Mean	-5.47 AB	-5.11 AB	-3.81 B	-5.39 AB	-3.87 B	-5.75 A
	SD	2.24	0.68	2.08	0.47	1.63	0.47
Ultrafil Firmset	Mean	-7.84 A	-5.93 B	-5.34 C	-5.17 CD	-4.70 D	-5.29 C
	SD	0.66	0.21	0.28	0.57	0.56	0.61
Ultrafil Regular Set	Mean	-5.51 B	-6.66 A	-6.05 AB	-6.58 A	-6.44 AB	-6.48 AB

	SD	0.73	1.01	1.25	0.64	0.43	0.31
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n = 10; \* = no beta-phase enthalpy peak for that particular run; negative values represent endothermic values

Groups with the same letter are statistically similar (Games-Howell, Ryan-Einot-Gabriel-Welsch Multiple Range Test;  $p = 0.05$ )

**Table 3. Alpha-Phase Mean Thermal Enthalpy (J/g)**

Material	RUN	A1	B1	A2	B2	A3	B3
Calamus	Mean	*	-7.52 B	-7.84 A	-7.62 B	-7.91 A	-7.61 B
	SD		0.05	0.08	0.2	0.07	0.1
DiaDent	Mean	*	-3.62 A	-3.74 A	-3.88 A	-3.88 A	-4.15 A
	SD		1.3	1.3	1.4	1.4	1.5
Elements HB	Mean	-3.46 A	-5.87 A	-5.72 A	-5.53 A	-6.03 A	-5.70 A
	SD	4.0	0.4	0.4	2.0	0.4	2.0
EndoRez	Mean	-6.11 A	-0.92 C	-4.15B	-4.73 AB	-5.36 AB	-5.49 AB
	SD	0.9	2.2	0.9	0.6	0.4	0.3
EndoSequence	Mean	*	-3.35 A	-3.63 A	-4.09 A	-3.77 A	-4.40 A
	SD		1.8	1.4	0.6	1.3	0.3
GuttaCore	Mean	-8.83 A	-3.30 C	-4.84 BC	-4.84 BC	-5.51 B	-5.36 B
	SD	3.2	0.9	0.6	0.7	0.6	0.7
K3	Mean	-7.792 A	-2.12 E	-4.30 D	-5.17 CD	-5.76 BC	-6.31 B
	SD	1.3	1.5	0.3	0.3	0.08	0.7
Lexicon	Mean	-1.16 D	-2.95 B	-2.53 C	-3.45 AB	-3.09 B	-3.78 A
	SD	0.4	0.4	0.3	0.4	0.3	0.4
Microseal	Mean	-12.85 A	-6.66 B	-0.81 C	-6.48 B	-7.58 B	-7.52 B
	SD	0.5	0.5	2.5	0.4	2.7	0.4
Obtura	Mean	-4.25 A	-4.34 A	-4.76 A	-4.62 A	-4.93 A	-3.29 A
	SD	1.8	0.5	0.4	0.4	0.4	3.3
Obtura Flow	Mean	-0.68 A	-4.56 A	-2.45 A	-4.58 A	-1.59 A	-4.60 A
	SD	2.2	3.2	4.0	3.2	3.4	3.2
Real Seal	Mean	*	*	*	*	*	*
	SD						
Resilon	Mean	*	-0.77 C	-3.38 B	-6.76 A	-7.47 A	-7.23 A
	SD		2.5	2.2	0.8	0.7	2.3
Schein Accessory Points	Mean	-5.87 A	-0.24 D	-3.55 C	-4.09 BC	-4.89 AB	-5.11 A
	SD	0.2	0.8	1.9	0.4	0.3	0.5

Simplifil	Mean	-4.84 CD	-5.02 C	-4.47 D	-5.54 AB	-5.28 BC	-5.98 A
	SD	0.7	0.3	0.5	0.4	0.4	0.5
SpectraPoint	Mean	-5.93 A	-1.63 B	-2.42 B	-2.14 B	-3.57 AB	-3.62 AB
	SD	1.8	2.6	2.3	2.3	1.9	1.6
SmartEndo Medium	Mean	-1.61 B	-3.21 AB	-4.60 A	-4.68 A	-5.27 A	-4.62 A
	SD	4.0	2.1	0.8	0.6	0.3	1.6
Thermafil Plus	Mean	-6.84 A	-3.43 B	-5.45 AB	-5.33 AB	-6.03 A	-5.87 AB
	SD	4.8	0.6	0.4	0.7	0.5	0.6
Successfil	Mean	-2.93 B	-5.68 B	-5.79 B	-5.06 B	-5.93 B	-5.14 B
	SD	0.1	0.8	2.1	1.2	0.3	0.3
Ultrafil Endoset	Mean	-6.27 A	-4.69 A	-3.63 B	-4.47 AB	-5.08 AB	-4.31 B
	SD	2.1	0.59	1.98	0.28	2.11	0.58
Ultrafil Firmset	Mean	-3.71 B	-4.63 A	-4.50 A	-4.52 A	-4.68 A	-4.50 A
	SD	0.5	0.1	0.1	0.1	0.4	0.1
Ultrafil Regular Set	Mean	-3.79 A	-3.01 AB	-3.19 AB	-2.96 AB	-2.69 B	-2.62 B
	SD	0.4	1.1	1.3	0.6	0.2	0.2

n = 10; \* = no alpha-phase enthalpy peak for that particular run; negative values represent endothermic values

Groups with the same letter are statistically similar (Games-Howell, Ryan-Einot-Gabriel-Welsch Multiple Range Test; p = 0.05)

## Discussion

Commercially-available gutta-percha for endodontic purposes is a viscoelastic polymeric material. This polymeric material, depending on the specific product, has been reported to consist largely of zinc oxide (66 to 84 percent) while the gutta-percha component ranges from approximately 14 to 20 percent.<sup>3</sup> This wide range of component variance presents some difficulty in comparing different commercial endodontic gutta-percha products as thermal and mechanical properties of these different materials depends on a great extent to the percentage composition of the mixture as well as the nature of the mixture components.<sup>14</sup>

The accepted thought concerning the existence of either  $\alpha$ - or  $\beta$ -form of gutta-percha depends on rotary motion of carbon units about isoprene units. These rotary motions cannot occur freely but exist as oscillations between two if not three preferred positions that are imposed by stereochemistry limitations due to valence bonds on adjacent carbon atoms.<sup>4</sup> In the more crystalline or ordered state at room temperature, these oscillations are thought not to occur freely and thus the stereochemical limitation position identifies the chain as either alpha or beta.<sup>4</sup> The as-received crystalline form, either  $\alpha$  or  $\beta$ , depends on the thermal stability of the specific commercial mixture. According to the established knowledge concerning dental gutta-percha, all of the samples tested as received should most likely have

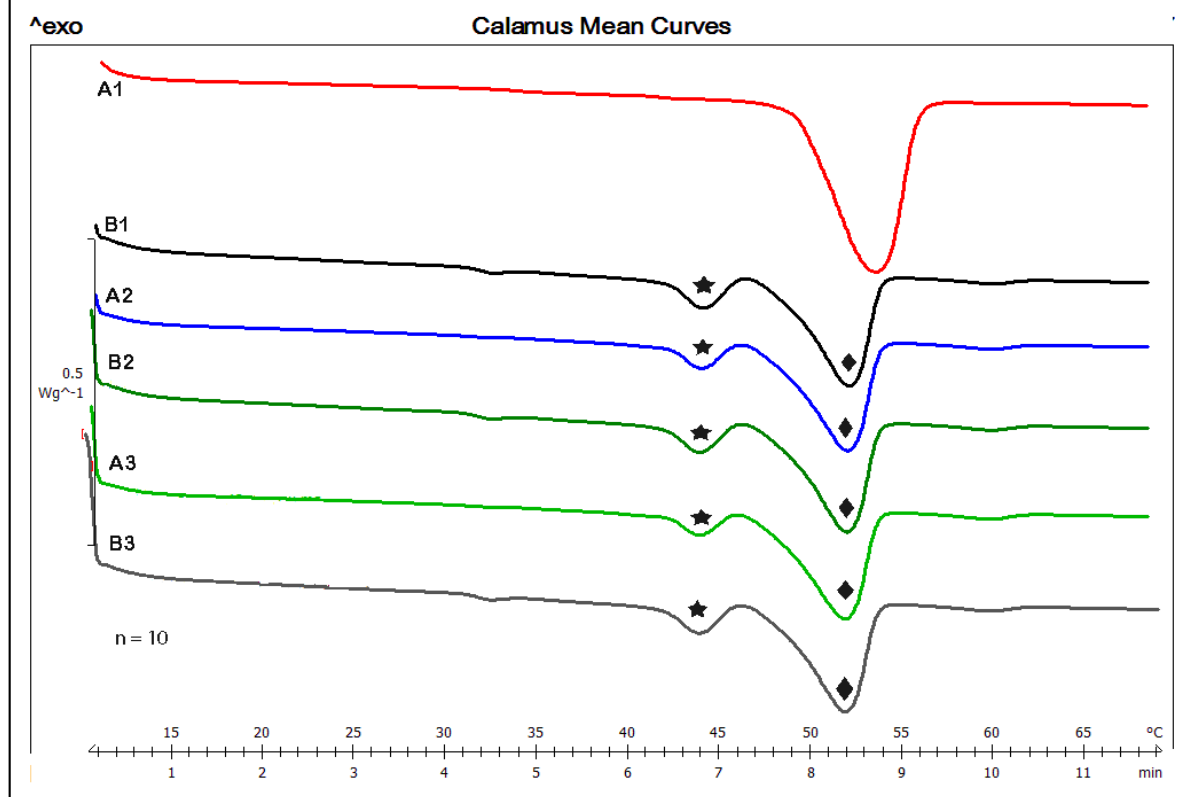
existed in a somewhat amorphous state with both  $\alpha$ - and  $\beta$  phases present. A rapid temperature rise above 70 °C should cause liquefaction and then the rapid cool should favor the formation of a predominately  $\beta$ -phased material.<sup>4</sup> Scientific investigations to date are largely in consensus that commercial endodontic gutta-percha formulations largely exist in the  $\beta$  semicrystalline state while some  $\alpha$  forms have been reported to exist at room temperatures.<sup>8,18</sup>

This study attempted to evaluate the stability of a number of commercially-available dental gutta-percha samples to elevated temperatures usually experienced during the vertical condensation obturation technique. The temperature that gutta-percha is subjected to with warm vertical condensation heat sources has been reported to vary and it is complicated further that the heating units themselves may be unreliable in attaining their set operating temperature.<sup>23-31</sup> These reports suggest that the gutta-percha rarely achieves the intracanal temperature as indicated by the heat source.<sup>23-31</sup> Nevertheless, this work was accomplished using the stated heat parameters since it has been reported from a recent study that 13 commercial endodontic gutta-percha formulations all demonstrated degradation at temperatures up to 130 °C.<sup>21</sup>

The thermal protocol was designed to evaluate the initial thermal signature of dental gutta-percha before and after a rapid rise in temperature followed by rapid cooling to room temperature. Any abrupt change in thermal enthalpies different than that usually experienced could signify an alteration in both the chemical and physical composition of the gutta-percha product, as thermal analysis has been demonstrated as a reliable analysis tool for gutta-percha.<sup>8,10,13,14,16,18,22,32</sup> The recommended temperature for the System B heat source is approximately 200 °C,<sup>33</sup> and since intracanal temperatures of gutta-percha are usually lower than the thermal source,<sup>23-29</sup> a rapid temperature rise to 230 °C was chosen to represent a worse-case scenario. The rapid cooling after the temperature rise was obtained by removing the aluminum DSC crucible to room temperature; although this does not exactly replicate the *in vivo* environment, this method was chosen as it could reliably effect the same cooling rate for all samples. After this first thermal protocol, the gutta-percha sample was analyzed thermally from 25 to 70 °C at a rate of 5 °C per minute. To assess the effect of reheating on the gutta-percha stability, this protocol was followed two more times.

The majority of the materials evaluated followed a similar basic pattern, as typified by the Calamus material mean results as displayed in Figure 25.

**Figure 25. Calamus Mean Enthalpy Curves**



With the First (A1) run, the thermal enthalpy signature was usually a broad endothermic peak ranging from 40 to 60 °C. The following runs demonstrated more definitive endothermic peaks typically associated with the lower-temperature  $\beta$ - to  $\alpha$ - transformation phase (denoted by star figures) and the higher-temperature  $\alpha$  semi-crystalline to an amorphous  $\alpha$  phase (denoted with diamond figures).

The first broad peak can be perceived as the materials demonstrating a more amorphous arrangement in the as-received condition. Although most commercially-available gutta-percha formulations are thought to be largely existing in the  $\beta$  semi-crystalline phase,<sup>8,18</sup> gutta-percha can also exist, depending on manufacturing methods, as a mixture of both  $\beta$ - and  $\alpha$  semi-crystalline states, of which the preponderance state will determine its physical characteristics.<sup>4</sup> Naturally-occurring gutta-percha exists in the  $\alpha$  configuration, and if it is heated and cooled at a rate above 0.5 °C/h it will be converted into  $\beta$  semi-crystalline state.<sup>7</sup> However, it should be understood for optimal conversion to the  $\beta$  semi-crystalline phase requires some time, and faster cooling rates may therefore "freeze" some  $\alpha$  semi-crystalline phase in the final product.<sup>4</sup> Some products also initially demonstrated an endothermic enthalpy peak between 60 and 62 °C. This curve was also identified in some gutta-percha products by Ferrante *et al.*<sup>22</sup> and is thought to be due to substances added during the manufacturing process. This report also confirmed earlier reports,<sup>22</sup> except for three products Elements HB, Lexicon, and Spectrapoint.

Commercially-available gutta-percha products represent a widely heterogeneous mixture,<sup>3,9,13,15</sup> and the various components may be compositionally randomly arrayed which would also produce a wide thermal enthalpy curve.<sup>18</sup> The subsequent thermal analysis challenges could increase the internal order of the material by possible removal of some of the more volatile components during the high-temperature challenge (waxes, etc.) and provide an annealing reorganization effect. In addition, it has been reported that commercially-available gutta-percha cones may undergo some change after manufacture, that was first thought due to oxidative processes<sup>34</sup> but was later reported due to transformation of the gutta-percha polymer to a more crystalline form.<sup>35</sup> Although current manufacturing methods and expedient shipping and storage conditions presumably have made these changes negligible, some small changes of the gutta-percha polymer cannot totally be excluded.

In addition to Calamus, a number of products demonstrated this first broad peak behavior. Some authors<sup>18</sup> suggest that these materials, as received, could demonstrate behavior closer to the  $\alpha$  semi-crystalline phase. The  $\alpha$  semi-crystalline phase is associated with less shrinkage upon heating than the  $\beta$  phase and is possibly thought to provide a better apical seal.<sup>12</sup> However, this observation should be interpreted with caution, as the proprietary manufacturing processes involved with these gutta-percha products are not fully known<sup>4</sup> and the overall enthalpy effects of the product's various combined constituents have not been definitively determined.<sup>18</sup> The definitive determination of the gutta-percha phase of the as-received products could be determined by X-ray diffraction,<sup>10,13</sup> which was beyond the scope of this evaluation.

Products demonstrating this thermal behavior on the first thermal challenge included Diadent, Elements HB, EndoRez, EndoSequence, Gutta Core, K3, Microseal, Obtura, SmartEndo, and Thermafil. Regardless of the as-received semi-crystalline phase, after the first high-temperature challenge and rapid cooling, the majority of gutta-percha based materials demonstrated thermal behavior typically observed with the two thermal phases of gutta-percha. Accordingly, Calamus, Diadent, Elements HB, EndoSequence, Gutta Core, K3, Obtura, SmartEndo, and Thermafil reverted to  $\beta$ - and  $\alpha$ - thermal enthalpy behavior after the first high-heat thermal challenge. More importantly, upon subsequent high-temperature challenge, the enthalpy peaks associated with both phases appeared to be stable with no appreciable degradation with these materials.

EndoRez is a novel gutta-percha material that is advertised to contain a dimethacrylate resin coating,<sup>36</sup> that is used with a self-priming resin sealer, and has been commercially available since the early 2000's.<sup>37</sup> EndoRez was found to have a solitary higher-temperature enthalpy peak with the first thermal cycle, which suggested a possible largely  $\alpha$  semicrystalline phase in the as-received form. Interestingly, after the first high-temperature challenge an intermediary solitary thermal signature was found whose temperature peak suggested more of the  $\beta$  phase but also exhibited a shoulder on the thermal curve suggesting some  $\alpha$  content remaining. The following thermal challenges showed EndoRez demonstrating typical  $\beta$ - and  $\alpha$ -phase thermal behavior. Interestingly, the accessory points from Henry Schein and Endorez demonstrated almost similar thermal behavior.

Microseal demonstrated thermal signatures suggesting possible  $\alpha$ -phase gutta-percha through three thermal cycles before  $\beta$ - and  $\alpha$ -phase stability was noted. One material that did not demonstrate this behavior and appeared to exist largely in the  $\beta$  semi-crystalline phase was Ultrafil Regular Set.

This evaluation also included two, non-gutta-percha obturation materials, Real Seal and Resilon, which are both mainly polyester materials. As can be seen in Figures X and X, both materials demonstrated essentially identical thermal signatures during the first two thermal challenges. After the third thermal run Resilon demonstrated two enthalpy peaks which suggested a possible change within the material. However, the two new peaks had stable enthalpies during the remainder of the thermal runs.

Even with noted enthalpy changes, it should be stressed that nearly all materials did not decompose and demonstrated thermal indications of stability in spite of the thermal challenges (up to 230 °C) that will exceed the *in vivo* temperatures experienced with warm gutta-percha techniques.<sup>24-29</sup> Although Ferrante *et al* reported additional physical changes of some gutta-percha products when heated above 130 °C,<sup>22</sup> no real thermal changes were noted under the conditions of this study.

The only exception noted under the conditions of this evaluation was Gutta Flow 2. Gutta Flow 2 is a combination of a polymethyldisiloxane sealer and particulate gutta-percha that are said to be less than 30 microns.<sup>38</sup> Gutta Flow 2 is used with a single master cone technique and is used as part of a cold obturation technique.<sup>38</sup> As can be seen in Figure X, this evaluation confirmed the manufacturer's usage recommendations as Gutta Flow 2 was observed to decompose after the first high-heat thermal challenge and should not be considered for use with any thermal obturation techniques.

#### Limitations:

1. Temperature cooling was to room temperature, not to relative intra-oral canal temperature, which would have been difficult to rapidly replicate with the established DSC technique;
2. Due to complex mixture of gutta-percha, thermally-derived definitive phase identification of as-received materials is difficult without X-ray diffraction.

#### Conclusions

1. Commercial forms of endodontic gutta-percha obturation materials overall demonstrated thermal characteristics that was material dependent;
2. Selected gutta-percha products that demonstrated thermal behavior first normally associated with the  $\alpha$  semi-crystalline phase reverted to  $\beta$  semi-crystalline phase after the first thermal challenge;
3. The majority of materials demonstrated stability at temperatures normally experienced during warm vertical condensation techniques. The only material that underwent decomposition under the conditions of this evaluation is not recommended for warm obturation techniques.

#### Disclosure

None of the authors have any commercial interests in any of the products or processes used during this evaluation. Their inclusion does not constitute endorsement. Any opinions expressed in this work are of the authors only and does not represent the official opinion of the United States Air Force, Department of Defense, or the United States Government.

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